

REMARKS

At the outset, applicants and their attorneys wish to thank Examiners Fordé and Flynn for the courtesy of the recent interview. The Examiners' careful attention to the application on that occasion is greatly appreciated.

The sole issue remaining to be resolved in this case is the rejection of the claims subject to examination as allegedly being based on an insufficient written description, which in turn is based on the Examiner's belief that the correction of the units of  $m^{-3}$  incorrectly set forth in the specification as filed to the correct units of  $cm^{-3}$ , constitutes an impermissible introduction of new matter into the present application.

The issue was discussed at the recent interview, at which time the Examiners expressed concern that the present record might not adequately support applicants' position that the correction does not constitute new matter. This paper therefore provides additional discussion and evidence in response to that observation, and which is believed to establish beyond question that the correction to the specification and claims effected by the amendment of November 4, 2002 merely makes corrections that would be manifest to those skilled in the art, and hence does not introduce new matter into the application.

In the present case, the units of  $cm^{-3}$  are set forth correctly in the priority application, Japanese Application No. 2000-085198, a point that can be confirmed by inspecting the

Japanese-language document already in the USPTO file. Therefore, there is no question from the present record that the units of  $\text{cm}^{-3}$  were set forth incorrectly in the present application as initially filed, as a result of mere typographical and/or translation error.

Under those circumstances, the case of *In re Oda*, 443 F.2d 1200, 170 USPQ 260 (CCPA 1971) establishes that correction of such errors will not constitute new matter if the skilled artisan (1) would have recognized that the original disclosure was mistaken, and (2) would have known what the correct disclosure should have been.

We note as an initial matter that neither the Official Action of January 28, 2003 nor the Examiner Interview Summary Record of mailed May 7, 2003 provides any evidence or reasoning in support of the allegation that the correction in question constitutes new matter. That is, no factual basis is offered in support of the conclusion that correction of the incorrect  $\text{m}^{-3}$  units to the correct  $\text{cm}^{-3}$  units does not merely reflect how a person skilled in this art would have naturally read the specification as filed. As such, the rejection as stated is insufficient as a matter of law.

Nevertheless, applicants wish to expedite favorable resolution of this matter, and therefore provide herewith further discussion and evidence on the substance of the question, as follows:

1. In all of the prior art documents of record, impurity dopant concentrations per unit volume are expressed as a given number of atoms per cubic centimeter, not per cubic meter, which evidence establishes the convention used by those skilled in the art.

2. A cubic centimeter is of course only one-millionth of a cubic meter, such that a given numerical value expressed per cubic meter would differ by six orders of magnitude from the same value expressed per cubic centimeter.

3. The claim recitation associated with the units of  $\text{cm}^{-3}$  pertains to an impurity concentration of "less than  $1 \times 10^{18} \text{ cm}^{-3}$ ." If the initially incorrect recitation of less than  $1 \times 10^{18} \text{ m}^{-3}$  were taken literally, this would translate to an impurity concentration of less than  $1 \times 10^{12} \text{ cm}^{-3}$ , an impossibly and absurdly low figure in the context of the invention.

4. The impurity concentration of the present invention is exemplified with reference to InGaN layers of a MQW structure, which are either lightly doped with Si, or undoped (unintentionally doped).

5. The attached page 135 from NAKAMURA (Ed.), *Introduction to Nitride Semiconductor Blue Lasers and Light Emitting Diodes*, shows that lightly doped GaN films are shown to have a carrier concentration of about  $10^{17} \text{ cm}^{-3}$ , whereas undoped (unintentionally doped) GaN films are known to have a carrier concentration of about  $10^{16} \text{ cm}^{-3}$ .

6. The attached GÖTZ et al. article shows that unintentionally doped GaN layers (undoped layers) nevertheless have a native Si concentration of the same  $10^{17} \text{ cm}^{-3}$  order of magnitude reported in the NAKAMURA textbook, owing to contamination from the components of the MOCVD apparatus. See, e.g., Table 1 at p. 3145 of the GÖTZ et al. article, wherein Sample No. 1 is not intentionally doped with Si.

7. Thus, the skilled artisan plainly would have recognized the initial recitation of "less than  $1 \times 10^{18} \text{ m}^{-3}$ " to be in error, as this would reflect a silicon concentration more than four orders of magnitude less than that known to be present in GaN layers when silicon is not even intentionally introduced. The skilled artisan would have furthermore known that the recitation should correctly have been --less than  $1 \times 10^{18} \text{ cm}^{-3}$ --, because, as noted above, (1) these are the units invariably used in practice in this art, and (2) these are the units in connection with which the numerical values make technical sense in the context of the invention.

From the above discussion, therefore, it is believed to be plainly and indisputably apparent that the correction of units effected by the amendment of November 4, 2002, does not introduce new matter into the application, and that the evidence of record now more clearly establishes that the skilled artisan (1) would have recognized that the original recitation of units as being in

YAMAGUCHI et al. S.N. 09/816,754

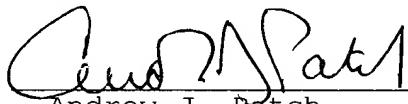
error, and (2) would have known what the correct units should have been

In view of the recent interview and the present amendment and the foregoing remarks, therefore, it is believed that this case is now in condition for allowance. Allowance and passage to issue are accordingly respectfully requested.

Respectfully submitted,

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# Activation energies of Si donors in GaN

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The electronic properties of Si donors in heteroepitaxial layers of GaN were investigated. The *n*-type GaN layers were grown by metalorganic chemical vapor deposition and either intentionally doped with Si or unintentionally doped. The samples were evaluated by variable temperature Hall effect measurements and photoluminescence (PL) spectroscopy. For both types of samples the *n*-type conductivity was found to be dominated by a donor with an activation energy between 12 and 17 meV. This donor is attributed to Si atoms substituting for Ga in the GaN lattice ( $\text{Si}_{\text{Ga}}$ ). The range of activation energies is due to different levels of donor concentrations and acceptor compensation in our samples. The assignment of a PL signature to a donor–acceptor pair recombination involving the Si donor level as the initial state of the radiative transition yields the position of the optical Si donor level in the GaN bandgap at  $\sim E_c - (22 \pm 4)$  meV. A deeper donor level is also present in our GaN material with an activation energy of  $\sim 34$  meV which is tentatively assigned to oxygen donors substituting for nitrogen ( $\text{O}_N$ ). © 1996 American Institute of Physics. [S0003-6951(96)03522-X]

The III–V nitride semiconductors, AlGaN, have evolved into the materials of choice for the fabrication of visible light emitting diodes exhibiting external quantum efficiencies approaching 10%.<sup>1,2</sup> Controlled *n*-type conductivity is commonly achieved by the incorporation of Si. Si substitutes for Ga in the GaN lattice and acts as a single donor.<sup>3</sup> Despite significant progress, little quantitative information is yet available on the electronic properties of Si donors. Also, the nature of the *n*-type background conductivity observed in most of the epitaxial GaN films is still not fully understood.<sup>4,5</sup>

Si donors in GaN were mainly characterized by variable temperature Hall effect measurements and donor activation energies of  $\sim 27$  meV are reported in the literature.<sup>6,7</sup> However, these values were derived from Arrhenius analyzes of the measured electron concentration with the implicit assumption of a temperature independent prefactor and, therefore, have a large uncertainty.

To clarify the situation for Si donors in GaN and to investigate the nature of the donors in our unintentionally doped material, we investigated *n*-type GaN films which were either unintentionally doped or Si doped by variable temperature Hall effect measurements and low-temperature (2 K) photoluminescence spectroscopy (PL). The Si content was determined by secondary ion mass spectrometry (SIMS).

The GaN films used in this study were grown by metalorganic chemical vapor deposition (MOCVD) on polished (0001)-oriented sapphire crystals. The unintentionally doped GaN film (sample No. 1) was  $\sim 4.5 \mu\text{m}$  and the Si-doped films (samples Nos. 2–5) were between 2 and 3  $\mu\text{m}$  thick. Sample No. 1 was grown in a different MOCVD reactor than samples Nos. 2–5.

The Hall effect measurements were conducted in the temperature range from 80 to 500 K. The magnetic field was

17.4 kG. Samples of  $5 \times 5 \text{ mm}^2$  size were cut from the wafers and metal dots were vacuum evaporated in the four corners to obtain electrical contacts in the Van der Pauw geometry.

For the PL measurements, the samples were mounted in a cryostat and immersed in pumped, liquid He to achieve a sample temperature of  $\sim 2$  K. The PL spectra were excited with the 325 nm line of a 30 mW HeCd laser with an incident power density of  $\sim 1 \text{ W/cm}^2$ . The resolution of the spectra was 0.05 nm.

Figure 1 presents results from variable temperature Hall effect measurements on an unintentionally doped GaN sample (No. 1). Figure 1(a) shows the electron concentration as a function of reciprocal temperature, and Fig. 1(b) the electron mobility as a function of the temperature. The electron concentration  $n$  [Fig. 1(a)] was derived from the experimental Hall constants  $R_H$  by:

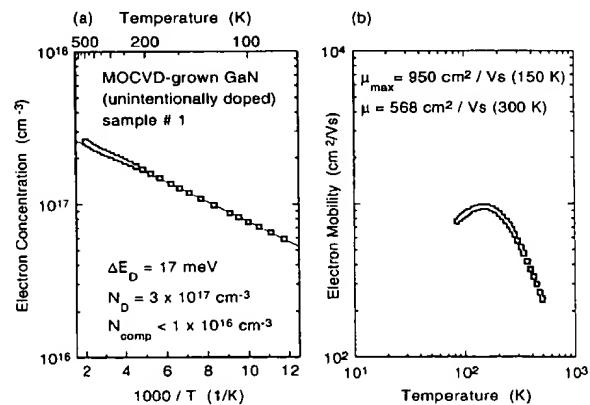


FIG. 1. Electron concentration vs reciprocal temperature (a) and Hall mobility vs temperature (b) for unintentionally doped, *n*-type GaN. The solid squares refer to the experimental data. The solid line in (a) results from a least-squares fit which is described. The fit yields parameters for a shallow donor which are defined as follows:  $\Delta E_D$  is the activation energy for donor electrons and  $N_D$  is the concentration of the donors.  $N_{\text{comp}}$  is the concentration of compensating acceptors present in the sample.

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TABLE I. Parameters for shallow donors as determined from least-squares fits of the charge neutrality equation to the experimental Hall effect data and results from secondary ion mass spectrometry (SIMS) for *n*-type GaN samples (column one). The parameter set includes donor electron activation energies ( $\Delta E_{D1}$ , columns two and four) and concentration of shallow donors ( $N_{D1}$ , columns three and five) for two independent donors. Also given is the concentration of compensating acceptors ( $N_{\text{comp}}$ , column six). SIMS results for the Si content are summarized in column seven.

Sample no.	$\Delta E_{D1}$ (meV)	$N_{D1}$ ( $\text{cm}^{-3}$ )	$\Delta E_{D2}$ (meV)	$N_{D2}$ ( $\text{cm}^{-3}$ )	$N_{\text{comp}}$ ( $\text{cm}^{-3}$ )	[Si] ( $\text{cm}^{-3}$ )
1	17	$3.1 \times 10^{17}$			$< 1 \times 10^{16}$	$4 \times 10^{17}$
2	15	$1.1 \times 10^{17}$	37	$3.9 \times 10^{16}$	$3.2 \times 10^{16}$	$2 \times 10^{17}$
3	14	$2.3 \times 10^{17}$	34	$6.9 \times 10^{16}$	$< 1 \times 10^{16}$	$5 \times 10^{17}$
4	12	$7.4 \times 10^{17}$	32	$6 \times 10^{16}$	$< 1 \times 10^{16}$	$9 \times 10^{17}$
5						$2 \times 10^{19}$

$$n = r_H(qR_H)^{-1}, \quad (1)$$

where  $r_H$  is the Hall scattering factor and  $q$  the electronic charge. Since  $r_H$  has not been measured for GaN it was assumed to be isotropic, temperature independent, and of unity value ( $r_H = 1$ ). In Fig. 1, the experimental data are represented by solid squares. The electron concentration in sample No. 1 gradually increases with temperature.

To obtain information about the donors which determine the electron freeze-out behavior of our unintentionally doped GaN, we performed a least-squares fit of the charge neutrality equation to the experimental data. The charge neutrality equation for *n*-type, wide band-gap semiconductors (intrinsic carriers are neglected) with  $M$  independent donors being given by:<sup>8</sup>

$$n + N_{\text{comp}} = \sum_{i=1}^M \frac{N_{Di}}{1 + \frac{g_i n}{N_C} \exp\left(\frac{\Delta E_{Di}}{kT}\right)}, \quad (2)$$

where the index  $i$  refers to the  $i$ th donor;  $N_{Di}$  is its concentration,  $g_i$  the degeneracy of its electronic state in the band gap ( $=2$ ), and  $\Delta E_{Di}$  the thermal activation energy of the donor electrons.  $N_{\text{comp}}$  is the concentration of compensating acceptors and  $N_C$  is the conduction band effective density of states ( $=4.3 \times 10^{14} T^{1.5} \text{ cm}^{-3} \text{ K}^{-1.5}$ );  $k$  is the Boltzmann constant and  $T$  the absolute temperature. Fit parameters are  $\Delta E_{Di}$ ,  $N_{Di}$ , and  $N_{\text{comp}}$ . For the fits, an effective electron mass of  $0.2m_0$  ( $m_0$  = mass of a free electron) was assumed. In Fig. 1(a), the calculated electron concentration as a function of the sample temperature is indicated by the solid straight line. A single donor level describes the electron freezeout behavior and the parameters of the donor level are depicted in Fig. 1(a) and summarized in Table I.

Figure 2 shows Hall effect results for Si-doped GaN samples (Nos. 2–5). The  $\text{SiH}_4$  flux during growth was gradually increased from sample Nos. 2–5. Consequently, the electron concentrations increased accordingly as determined by the Hall effect measurements. For samples Nos. 2–4 the experimental Hall effect data were used to obtain information about shallow donors which give rise to the *n*-type conductivity in Si-doped GaN films by least-squares fits of Eq. (2). For these fits, two independent donor levels had to be employed. Defect parameters are summarized in Table I. The temperature dependence of the electron concentration of

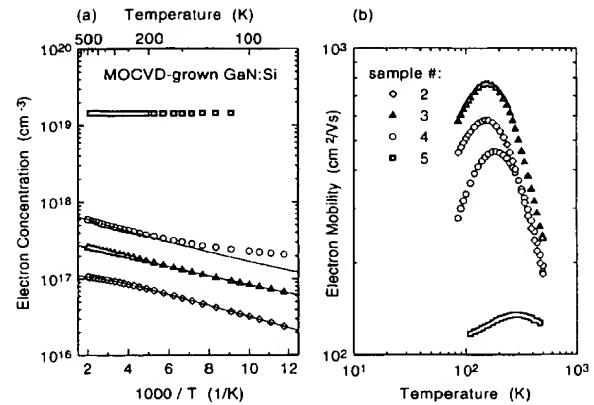


FIG. 2. Electron concentration vs reciprocal temperature (a) and Hall mobility vs temperature (b) for GaN doped with increasing Si concentration. The symbols refer to the experimental data. The solid lines in (a) result from least-squares fits to the experimental data. The fits yield parameters for shallow donors which are summarized in Table I.

sample No. 4 at temperatures below 250 K indicates defect band conduction leading to a deviation of the fitted curve from the experimental data. For sample No. 4 only data above 250 K were used for the fit. The electron concentration of sample No. 5 does not vary with the sample temperature indicating a doping level above the degeneracy limit. The room temperature and the peak mobilities of the Si-doped samples depend on the Si content. Sample No. 2, which has the lowest Si content, has a room-temperature (300 K) mobility of  $370 \text{ cm}^2/\text{Vs}$  and a peak mobility at 150 K of  $582 \text{ cm}^2/\text{Vs}$ . The highest mobilities for Si-doped material in sample No. 3 with values of  $501 \text{ cm}^2/\text{Vs}$  (300 K) and  $764 \text{ cm}^2/\text{Vs}$  (160 K). In sample No. 4 the mobilities are  $369 \text{ cm}^2/\text{Vs}$  and  $459 \text{ cm}^2/\text{Vs}$  (190 K). For sample No. 5 the peak mobility is measured at 300 K with  $135 \text{ cm}^2/\text{Vs}$ . The temperature dependence of the mobilities at temperatures higher than the peak mobilities is well approximated by a power dependence  $\sim T^{-1.5}$  for all Si-doped GaN films shown in Fig. 2(b) (except sample No. 5).

The Hall effect analysis shows that the *n*-type conductivity in our unintentionally doped material (sample No. 1) is due to a shallow donor level with an activation energy of  $\sim 17 \text{ meV}$  (Table I). A donor with a similar activation energy ( $\Delta E_{D1}$ ) is present in samples Nos. 2–4, which were intentionally doped with Si (Table I). The concentration of this donor ( $N_{D1}$ ) increases according to the flow rates of  $\text{SiH}_4$  which was increased from samples Nos. 2–5. Therefore, it seems reasonable to conclude that the shallow donor level with an activation energy in the range between 12 and 17 meV is due to Si incorporation into GaN and that the dominant donor in our unintentionally doped GaN is also Si. Results from secondary ion mass spectrometry (SIMS, Table I) support this conclusion. First, the concentration of Si as determined by SIMS increases according to the concentration of the shallowest donor level (Table I). Second, the atomic concentration of Si is found to be  $\sim 4 \times 10^{17} \text{ cm}^{-3}$  in sample No. 1 (Table I).

Our determination of the thermal activation energy for ionization of Si donors is based on an accurate analysis ac-

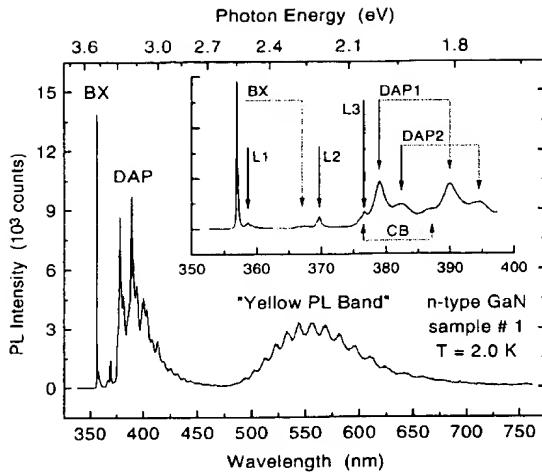


FIG. 3. PL spectrum for sample No. 1. PL emission lines are labeled "BX", "DAP", and "yellow PL band". The inset magnifies the high energy portion of the spectrum. Zero-phonon lines are indicated by arrows with solid lines and their first LO-phonon replicas by arrows with dotted lines. The arrows labeled "CB" indicate a recombination process that may involve the conduction band as the initial state.

cording to the charge neutrality condition, Eq. (2), and yields activation energies  $\Delta E_{D1}$  in the range between 12 and 17 meV. The decreasing activation energies with increasing donor concentrations observed for samples Nos. 2–4 (Table I) can be explained by interaction of the donor wave functions which reduces ionization energies, as has been observed for other semiconductors, e.g., Si and Ge.<sup>9</sup> However, the unknown Hall scattering factor and its temperature dependence pose an uncertainty in the determination of this value.

The activation energy for donor ionization cannot be determined from variable temperature Hall effect measurements by a slope analysis in a linear region of the electron concentration versus reciprocal temperature data, as demonstrated in Refs. 6 and 7. Such an analysis yields  $\sim 4/3$  of the actual activation energy as can be shown by a Taylor series analysis of Eq. (2).

A PL spectrum for sample No. 1 is shown in Fig. 3. The spectrum taken at 2 K, exhibits features which are generally observed for *n*-type GaN. The strongest line (BX) appears at 357.0 nm (3.473 eV) with a FWHM of  $\sim 2.2$  meV. This emission line is due to the annihilation of excitons bound to neutral shallow donors. At lower phonon energies a series of lines labeled DAP is present in the spectrum. In the literature (e.g., Ref. 10), they are usually referred to as due to donor–acceptor recombinations. The yellow luminescence band centered at  $\sim 2.2$  eV is also present in our material. The inset in Fig. 3 magnifies the high energy portion of the PL spectrum. The PL line labeled "L1" is positioned at 358.6 nm (3.458 eV) and is likely to be an acceptor bound exciton. The

lines "L2" and "L3" at 369.7 nm (3.353 eV) and 376.7 nm (3.292 eV), respectively, may indicate Mg contamination since these lines usually appear in our Mg-doped GaN; their origin is unknown. The emission lines labeled "DAP1" and "DAP2" are associated with donor–acceptor pair recombinations and are positioned at 379.0 nm (3.272 eV) and 390.0 nm (3.179 eV), respectively. A broad PL line at  $(376.4 \pm 0.4)$  nm  $[(3.294 \pm 0.004)$  eV] is labeled "CB".

In the following, we use the PL emission lines "CB" and "DAP1" to suggest the positions of the Si donor level and the Mg acceptor in the GaN band gap. DAP1 is the highest energy line and, therefore, should involve the shallowest donors and acceptors, namely Si and Mg. We assume that the broad line "CB" is due to the same radiative transition, however, with the initial state being the conduction band edge. The energy difference between the CB and the DAP1 line yields the position of the Si donor level at  $E_c - (22 \pm 4)$  meV. Consequently, the optical level for the Mg acceptors follows at  $E_v + (209 \pm 5)$  meV [ $E_{gap} = (3.503 \pm 0.0005)$  eV, 2 K].<sup>11</sup>

A second donor level with electron activation energies ( $\Delta E_{D2}$ ) between 32 and 37 meV is observed in samples Nos. 2–4 (Table I). This donor level may be due to oxygen contamination. Oxygen substituting for nitrogen has been suggested to act as a shallow donor and may contribute to the *n*-type background conductivity in unintentionally doped GaN.<sup>5</sup>

In conclusion, we have determined the activation energy for ionization of Si donors in GaN by variable temperature Hall effect measurements. For the samples investigated in this study, the analysis yields activation energies in the range between 12 and 17 meV. Therefore, and due to its high solubility in GaN Si is well suited as a donor dopant for the preparation of *n*-type GaN layers with electron concentrations in the range up to  $\sim 10^{20}$  cm<sup>-3</sup>.

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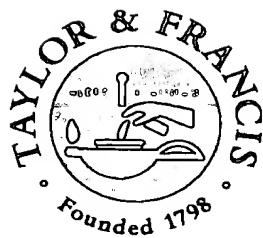
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# Introduction to Nitride Semiconductor Blue Lasers and Light Emitting Diodes

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The spatial variation of luminescence in smooth films of unintentionally doped GaN is shown in Figure 4.29. In contrast with the preceding case, the sapphire surface had not been exposed to ammonia. The rocking curve full-width at half-maximum was 6 arcmin. The secondary electron image in Figure 4.29a demonstrates the featureless planar morphology of the film. Light emission at the band-edge region ( $\lambda = 364\text{nm}$ ) in Figure 4.29b is characterized by a granular appearance, where the bright spots represent regions of high CL emission intensity, and dark spots represent low emission intensity. The bright spots are distributed with separation ranging between 0.3 and  $1.0\mu\text{m}$ . The smallest separation that is observed among features in this image is  $0.3\mu\text{m}$ . Light emission in the yellow luminescence band ( $\lambda = 364\text{nm}$ ) is shown in Figure 4.29c, where a granular structure is also observed but with smaller features than in the band-edge emission images.

The variation of luminescence along the thickness of the material can be observed by looking at a cross-section specimen, such as those prepared for transmission electron microscopy. Figure 4.30 is a CL image taken for  $\lambda = 364\text{nm}$ . Dark lines running longitudinally along the growth direction are observed. The morphology and density of these lines resemble the dislocation arrangement observed by TEM, for example in Figure 4.10.

These observations indicate that in undoped GaN films the yellow luminescence is spatially associated with the dislocation distribution. This leads to the hypothesis that the dislocations that are necessary to maintain local coherence in the columnar, low-angle domain boundaries, are associated with the yellow luminescence. The states in the gap that produce the deep-level (yellow) luminescence either arise directly from the atomic structure of the dislocations, or are associated with the clustering of negative point defects (vacancies, interstitials) or impurity atoms (such as oxygen and carbon) due to the strain and electrochemical lattice distortions produced by the presence of dislocations.

#### 4.8.2 Doped Materials

The inhomogeneous spatial variation of CL in lightly doped materials ( $\sim 10^{17}\text{ carriers/cm}^3$  at room temperature) is absent in undoped materials ( $\sim 10^{16}\text{ cm}^{-3}$ ). Figure 4.31 shows the surface morphology and CL characteristics of a GaN thin film doped with silicon ( $\sim 2 \times 10^{17}\text{ cm}^{-3}$ ), grown under the same conditions as the undoped film in Figure 4.29. The secondary electron image in (a) is indicative of a specular, featureless surface. The CL band-edge emission image in (b) shows spatially-homogeneous luminescence. The yellow luminescence in (c) is significantly weaker and exhibits a high degree of uniformity down to the sub-micron range. We have also observed similar uniformity in light emission in p-type (Mg-doped) GaN films.

The uniformity of the luminescence in lightly doped samples has some interesting implications about the role of dopants in the GaN lattice. It suggests some type of interaction between dislocations and the impurities in the neighbouring lattice. The following processes could be responsible for these observations: